Preliminary Study of Nitration of PETRONAS Calcined Cokes For Water Dispersible Solid Fuel

by

Hidayah Binti Awalludin

Dissertation submitted in partial fulfilment of

the requirements for the

Bachelor of Engineering (Hons)

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Mechanical Engineering Programme
Universiti Teknologi PETRONAS
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Approved by,

____
(AP Dr. Bambang Ari Wahjoedi)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

December 2009

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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HIDAYAH BINTI AWALLUDIN

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

PAGE

FIGURE

TABLE

ABSTRACT

The objectives of this work are to determine if the calcined coke can be water dispersible, to study the improvements of calcined coke after treating with nitration, and to compare the characteristics of solid fuel and nitrated calcined coke to see if there is a possibility for the coke to be developed to water dispersible solid fuel.

Currently calcined coke primary use is in making carbon anodes for aluminum industry. Because of the insoluble properties, the usage of calcined coke is limited. Plus, calcined coke has low volatile matter, high ash content and high fixed carbon content which make it unsuitable for solid fuel.

Since nitration can remove ash content and increase solubility of a compound, by having this project we can see how the properties of calcined coke can be improved after being treated with nitration process.

The keywords for this project are calcined coke, nitration, solid fuel, volatile matter, ash content, fixed carbon content and solubility.

ACKNOWLEDGEMENT

Thank you Allah the Almighty for His will enable me to complete this final year project. Here I want to express my gratitude to all the contributors whom helped me throughout this project. First and foremost, thanks to Allah for the blessing and the health given to me while working on this project throughout the semester.

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THANK YOU ALL.

CHAPTER 1
INTRODUCTION

1.0 INTRODUCTION

1.1 BACKGROUND STUDY

Petroleum coke is a black colored solid produced by the high pressure thermal decomposition of heavy or high boiling petroleum process streams and residues. Calcined petroleum coke has lower volatile matter, higher percentage of elemental carbon and lower potential for toxicity [1].

In the other hand, solid fuel refers to various types of solid material that are used as fuel to produce energy and provide heating, usually released through combustion. Solid fuels have long been used by humanity to create fire.

The use of some solid fuels is restricted or prohibited in some urban areas, due to unsafe levels of toxic emissions. The use of other solid fuels such as wood is increasing as heating technology and the availability of good quality fuel improves. In some areas, smokeless coal is often the only solid fuel used.

1.2 PROBLEM STATEMENT

1.2.1 PROBLEM IDENTIFICATION

The primary use of calcined coke is in making carbon anodes for aluminum industry. Since petroleum coke is insoluble, developing product of petroleum coke based is limited.

However, there is a possibility that these calcined cokes might be able to produce a water dispersible solid fuel after undergoing a certain treatment. That way, the application of calcined cokes can be developed.

1.2.2 SIGNIFICANCE OF PROJECT

The significance of this project is that in the future, Petronas, the biggest oil & gas company, would consider developing more of its side products from oil refinery process to something that can give benefits and profits to the company. Rather than being wasted, those side products might turn into something innovative if research and development conducted thoroughly.

This project is relevant to the study of Oil and Gas as well as the study of underground soil structures. This project is also relevant to maximize the usage of un-renewable energy which in this case is petroleum.

1.3 OBJECTIVES & SCOPE OF STUDY

The objectives of this work are to determine if the calcined coke can be water dispersible, to study the improvements of calcined coke after treating with nitration, and to compare the characteristics of solid fuel and nitrated calcined coke to see if there is a possibility for the coke to be developed to water dispersible solid fuel.

The scope of study involved would be the reactions of Petronas cokes towards nitration process which can be study by using Thermo Gravimetric Analysis and Fourier Transform Infrared Spectroscopy (FTIR Analysis).

CHAPTER 2
LITERATURE REVIEW

2.0 LITERATURE REVIEW

2.1 PETROLEUM COKES

Petroleum coke is carbonaceous solid derived from oil refinery coker units, which has been cracked or otherwise processed to remove low boiling fractions. Oil refinery is an industrial plant where crude oil is processed and refined into more useful petroleum products [2].

Coke is made by heat-treating the residual oil (more accurately described as tar) to a temperature high enough to result in its polymerization to form a non-melting solid carbon. The process is performed to maximize the yield of lower molecular weight compounds derived from crude oil feedstock. The composition varies depending on source of final product.

Petroleum cokes can be categorized generally as either green or calcined coke. The initial product of the coking process, green coke, is used as a solid fuel. Further processing of green coke at higher temperatures and pressures result in calcined coke which is used in the manufacture of electrodes, in smelting applications, for graphite electrode production, or for minor applications such as carbonization of steel.

Petroleum coke is characterized by its chemical composition and physical characteristics. The chemical composition of petroleum coke is dependent upon the composition of the feedstocks that are used in the coking process, which in turn are dependent upon the composition of the crude oil from which they are derived. The metals and sulfur composition of calcined coke is directly dependent upon the composition of the green coke from which it was produced.

The physical characteristics of petroleum coke are important in determining the suitability of a coke sample for a specific use [3].

2.1.1 CHARACTERISTICS OF CALCINED COKES

Calcined coke is a hard, dense substance with low hydrogen content, a low coefficient of thermal expansion, and good electrical conductivity. These properties, along with low metals and ash contents make calcined petroleum coke highly desirable for use in the aluminum smelting industry [4].

Typical parameters measured to define the chemical composition of petroleum coke are weight % ash, weight % sulfur, weight % residual hydrocarbon. Residual hydrocarbon includes organic matter ranging from 6-carbon compounds to 7-ring polycyclic aromatic hydrocarbons (PAHs). Because of the lower temperature used in its production, green or fuel-grade coke contains higher levels of residual hydrocarbon than other grades of coke [5].

Calcinations basically removed volatile carbon matter, hydrogen, residual hydrocarbon and some of sulfur and nitrogen present in green cokes. As a result, ash, fixed carbon and carbon contents increased, while calorific value decreased. Loss or gains of each coke property value after calcinations are compared in the following:

Table 2.1: Loss or gains of each coke property value after calcinations [6].

Property	**Loss, %**	**Gain, %**
Sulfur	8-13	
Ash		9-46
Carbon		5-6
VCM	95.4 – 97.6	
Residual Hydrocarbon	95 - 98	

2.2 SOLID FUEL (COKE FUEL)

2.1.1 CHARACTERISTICS OF SOLID FUEL

Some basic fuel properties, as well as the fuel response to specialized tests developed to identify fuel ignition and carbon burnout characteristics are essential to define the proper furnace configuration. Combustion characteristic indicators include the following:

Volatile Matter and Fixed Carbon Content

One of the most important fuel constituents is the volatile matter (VM) content. This constituent has a very large impact on the ignitability and burnout characteristics of solid fuels. VM is driven out of the fuel when the fuel is heated and ignites readily, thus supporting ignition of the fuel.

Fixed carbon (FC) is the other constituent in solid fuels that burns. FC is more difficult to ignite and burns more slowly than VM. As VM decreases, the ratio of the FC to VM increases. A fuel with a higher FC/VM ratio is more difficult to burn than a fuel with a lower ratio.

Regardless of the basis of measurement, the general rule is that as the VM decreases (and the FC/VM ratio increases) the fuel becomes harder to ignite and burns more slowly. VM content is often used as an indication of the ease of combustion of solids fuels [7].

Reactivity Index

VM alone was not sufficient for predicting the burning characteristics of solid fuels. While the general trend showed fuels with lower VM content were more difficult to burn than fuels with a higher VM content, there appeared to be a relatively large difference in burning characteristics for fuels of the same VM.

RI tends to increase significantly when the VM drops [8].

Table 2.2 : Comparison of Solid Fuel & Calcined Coke Properties

Properties	**Solid Fuel**	**Calcined Coke**
Volatile Matter	Volatile matter support fuel ignition	Has low volatile matter
Fixed Carbon	More difficult to ignite and burns slower than VM	High fixed carbon content
Reactivity Index	High reactivity index, harder to burn	High reactivity index because of low VM

Based on the table above, we can conclude that to be compatible as a solid fuel, the characteristics needed are high volatile matter, low fixed carbon and low reactivity index. However, calcined coke has nothing like the characteristics mentioned. Low volatile matter, high fixed carbon and high reactivity index make it almost impossible for calcined coke to be developed into solid fuel. Moreover, calcination removed almost entire hydrocarbon in calcined coke resulting to its insolubility.

2.3 NITRATION

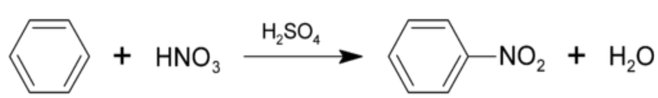
Nitration of hydrocarbons, particularly of aromatic compounds, is probably one of the most widely studied organic reactions. The classical nitration method usually requires the use of an excess of nitric acid and the assistance of strong acids such as concentrated sulfuric acid [9].

2.3.1 EFFECT OF NITRATION TO PETROLEUM COKE

An effect of nitration towards petroleum coke is de-ashing. There are indications that of the possibility of removing ash from carbonaceous materials by treating them with nitric acid.

Apparently, there is a simple aromatic hydrocarbon called Benzene. Benzene and the other aromatic hydrocarbons are obtained for industrial purposes from the distillation of coal tar, a byproduct in the manufacture of coke, and from petroleum. There is a possibility that Benzene exists in petroleum coke. By treating the petroleum coke with nitration, a nitro group will be introduced to the sample of petroleum coke.

Benzene is nitrated by refluxing with concentrated sulfuric acid and nitric acid.. The sulfuric acid is regenerated and hence acts as a catalyst. It also absorbs water. Below is the equation of nitration of benzene [10].



(1)

$$2H_2SO_4 + HNO_3 \longrightarrow 2HSO_4^- + NO_2^+ + H_3O^+ \quad (2)$$

$$C_6H_6 + NO_2^+ \longrightarrow C_6H_5NO^2 + H^+ \quad (3)$$

$$H^+ + H_3O^+ + 2HSO_4^- \longrightarrow H_2O + 2H_2SO_4 \quad (4)$$

The nitro group introduced to the compound substantially enhanced the solubility of petroleum coke in aqueous organic solvents. The existence of the nitro group can be detected using the Fourier transform infrared (FTIR) analysis [11].

Table 2.3: Properties of Calcined Coke After Nitration Process

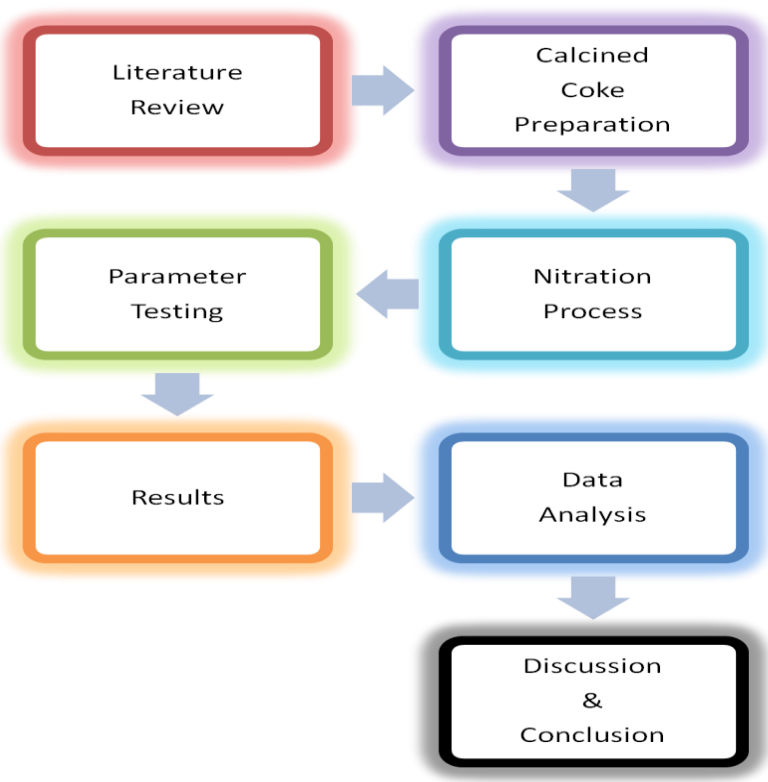
Properties	**Nitration**	**After-Effect**
Ash Content	Remove ash content	Low ash content, low reactivity, easier to burn
Solubility	Add nitro group into the compound	Increase solubility

CHAPTER 3
METHODOLOGY

3.0 METHODOLOGY

The whole project started with literature review. And after that is the process of preparing the calcined coke. The nitration process will take place after that. During the nitration process, three samples of calcined coke are treated with nitration at three different temperatures which are 50, 75 and 90˚C. These three samples of calcined coke along with the reference sample of calcined coke (untreated calcined coke) then underwent parameter testing which are FTIR Analysis and also Thermo Gravimetric Analysis. From the results gained, the analysis is done. After thorough analysis and discussion, then only the project concluded whether it’s successful or not.

Figure 3.1: Flowchart of the Project Progress



3.1 NITRATION

The main point of the project is the nitration of calcined cokes with three different temperatures which are 50, 75 and 90˚C. Below are the apparatus used in the process.

Apparatus:

1. Nitric Acid & Concentrated Sulfuric acid
2. Petroleum coke
3. Beaker
4. Stirrer/Glass rod
5. Thermometer
6. Heater
7. Filter
8. Hydrochloric Acid
9. Oven
10. Fume Cupboard
11. Distilled Water
12. Gloves

Procedure:

1. 12.5 g of pulverized petroleum cokes were added in small amount at a time into a reaction set up containing 250 mL of concentrated sulphuric and nitric acids.
2. The mixture were cooled down using a water bath while constant stirring were applied until the evolution of reddish brown gaseous nitric acid (HNO_2) was about to stop.
3. The mixture then heated for 5 hrs at different temperature i.e. 50, 75 and 95˚C with constant stirring.
4. The mixture then cooled to room temperature while maintaining the agitation and the nitrogen (optional) flow overnight.
5. The reaction mixture was then poured into 1250 cm^3 distilled water, filtered and washed with a continuous flow of distilled water until filtrate gave pH > 4.
6. The filtrate also checked for product in the solution by treating it with concentrated HCl solution.
7. The wet, nitrated petroleum coke weighed and then dried using oven at 60˚C [12].

The visualization of the nitration process

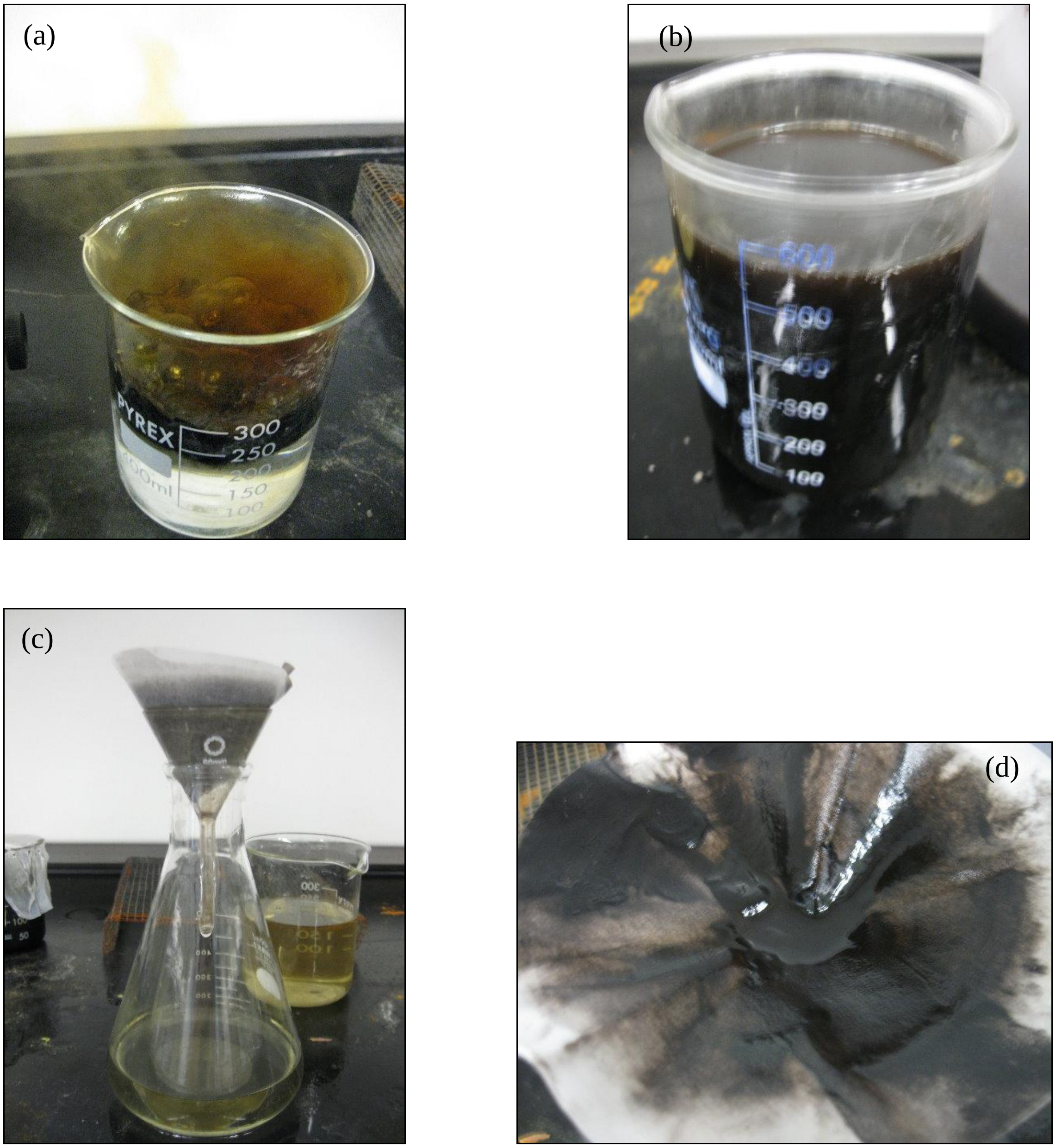


Figure 3.2: (a) Reddish brown gaseous evolved when concentrated HNO_2 + H_2SO_4 mixtured with calcined coke, (b) After 5 hours of heating, the mixture color changed to black, (c) The mixture filtered to separate the nitrated calcined coke from the solution, (d) Wet nitrated calcined coke.

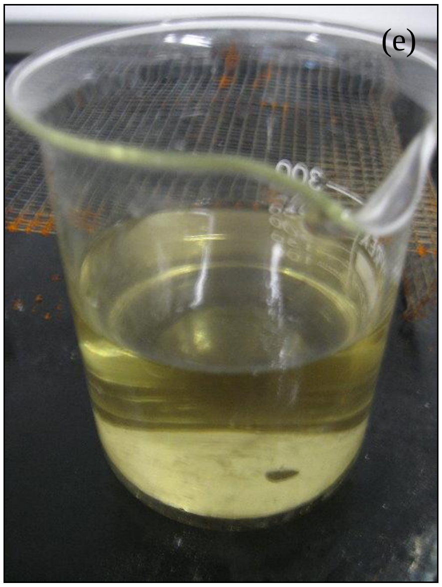


Figure 3.2(cont’d): (e) The remaining solution, (f) The nitrated calcined coke after baked in the oven.

CHAPTER 4

RESULTS & DISCUSSIONS

4.0 RESULTS

After the nitration process, there are significant differences in the sample's weight which showed below.

Table 4.1: Weight Comparison of Calcined Coke Before & After Nitration

Temperature of Treatment (℃)	**50**	**75**	**95**
Weight Before Nitration (g)	12.5	12.5	12.5
Weight of Wet Nitrated Calcined Coke (g)	24.267	28.363	23.659
Weight of Dry Nitrated Calcined Coke (g)	14.380	17.933	13.63
Weight Gained (g)	1.880	5.433	1.130

4.1 Thermo Gravimetric Analysis

The results from Thermo Gravimetric analysis reveal the weight percentage of the sample at different temperatures. The results are shown in the graphs below.

Figure 4.1: Weight % vs. Temperature Graph for Un-Nitrated Calcined Coke

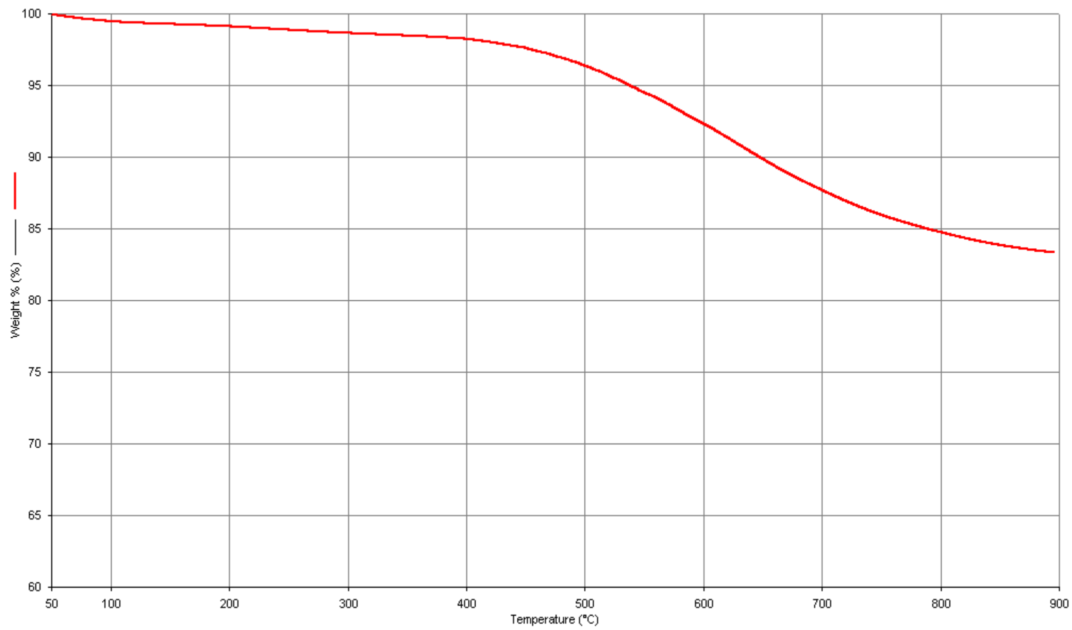


Figure 4.2: Weight % vs. Temperature Graph for Calcined Coke Nitrated at 50˚C

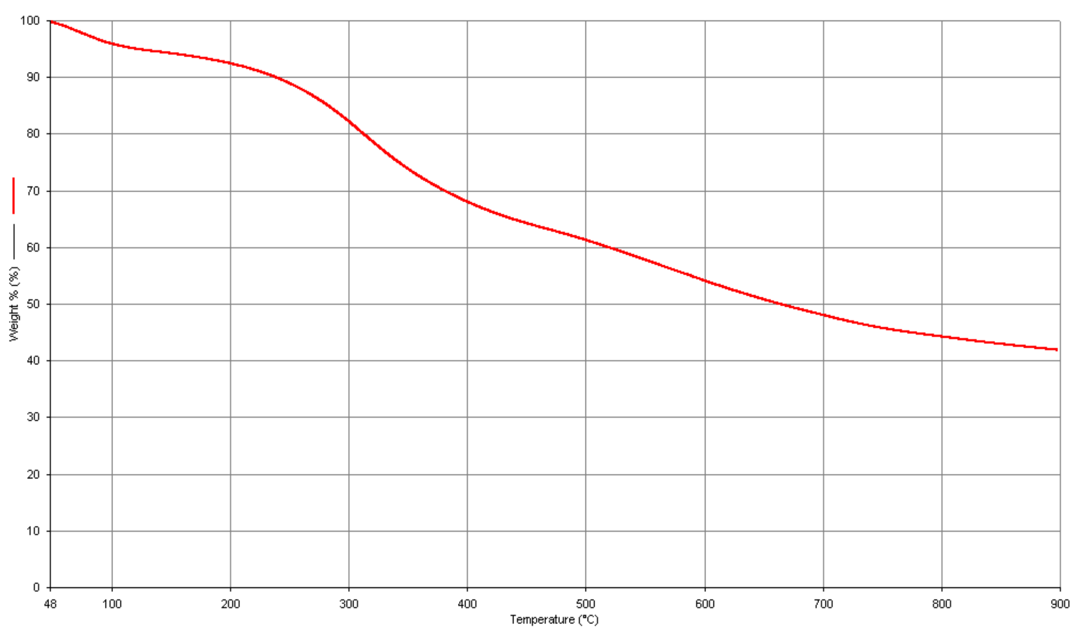


Figure 4.3: Weight % vs. Temperature Graph for Calcined Coke Nitrated at 75˚C

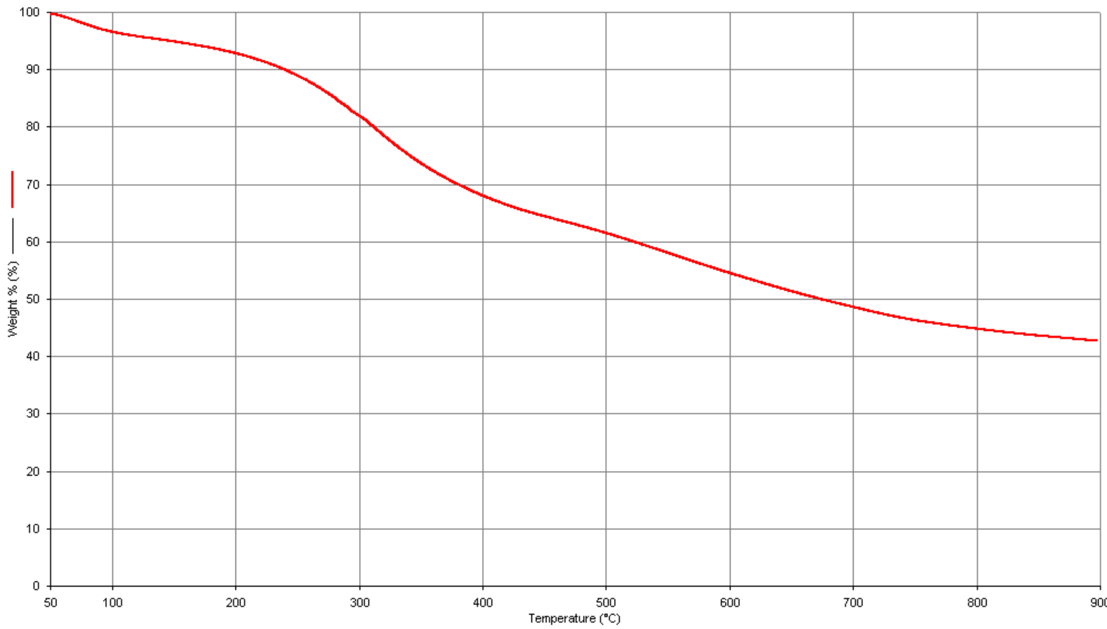
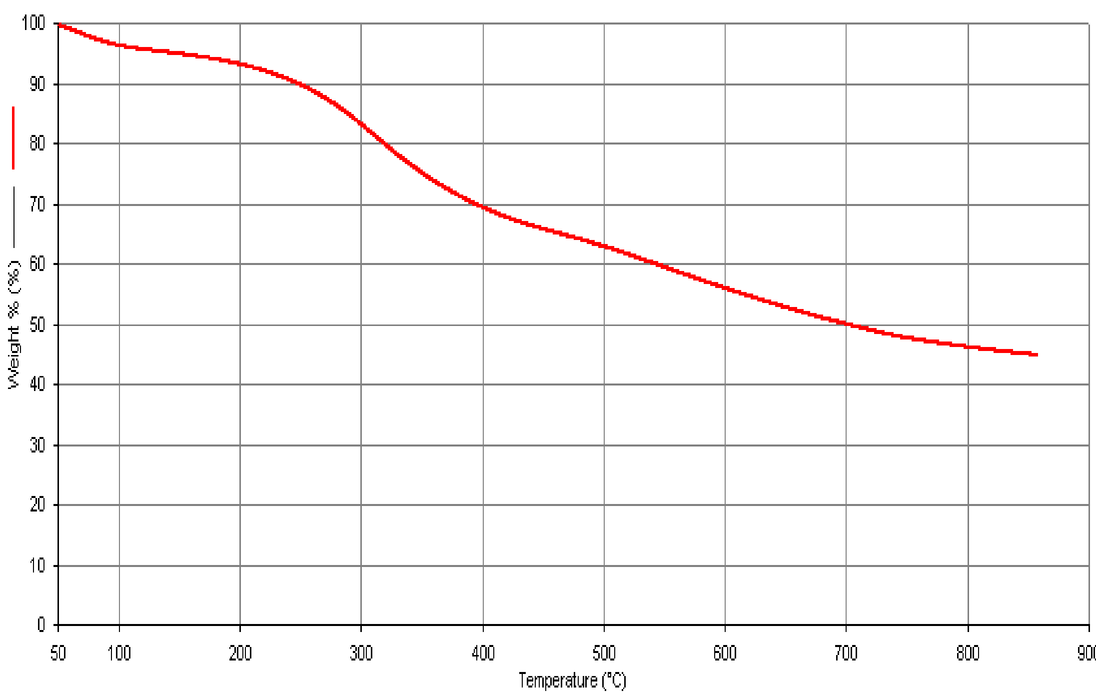


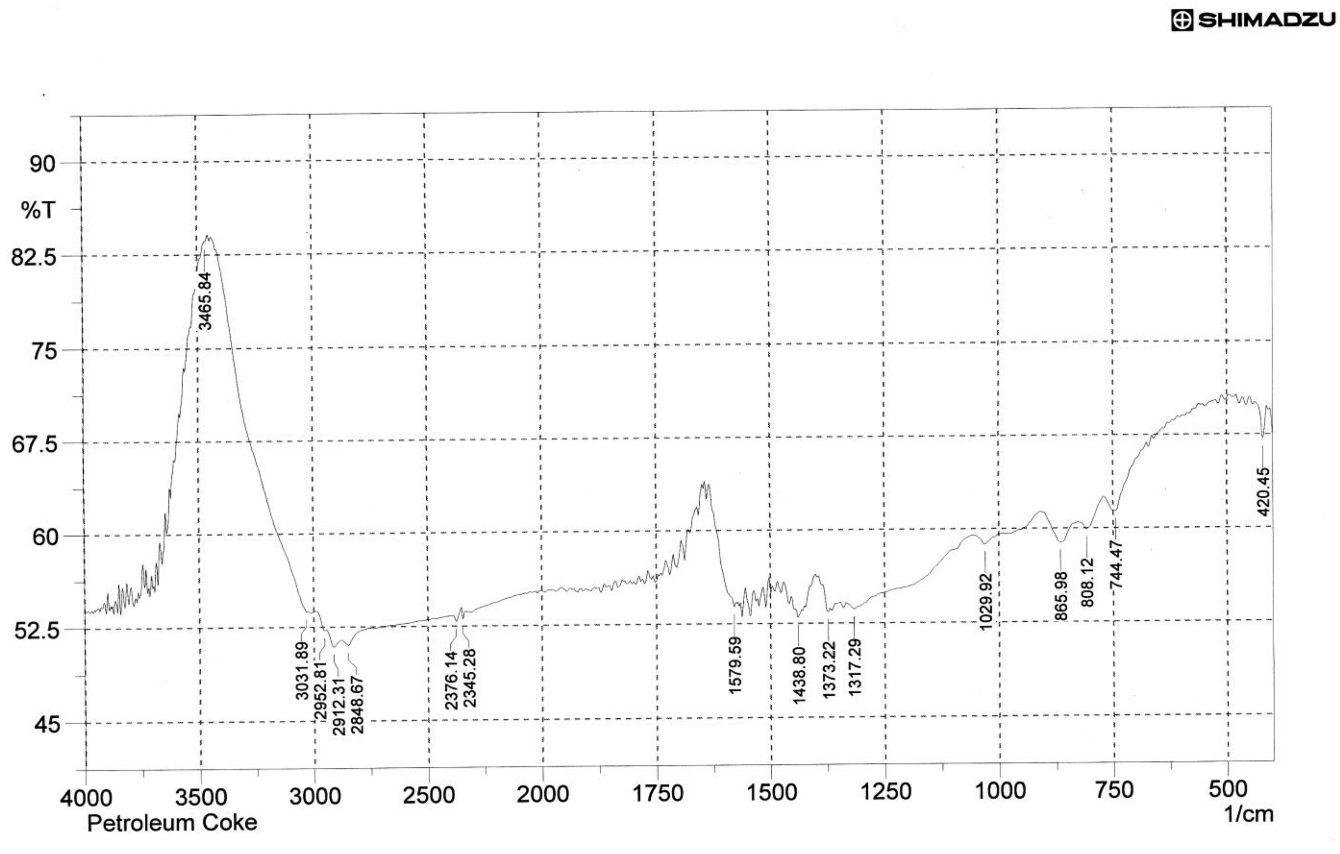
Figure 4.4: Weight % vs. Temperature Graph for Calcined Coke Nitrated at 95˚C



4.2 FTIR (Fourier Transform Infrared Spectroscopy) Analysis

The results from FTIR (Fourier Transform Infrared Spectroscopy) analysis shown the wave numbers of compounds in the sample and the percentage of transmittance. Each of the peak shown in the graph represent different compound or component in the sample.

Figure 4.5: FTIR Spectrum for Untreated Calcined Coke



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Petroleum Coke

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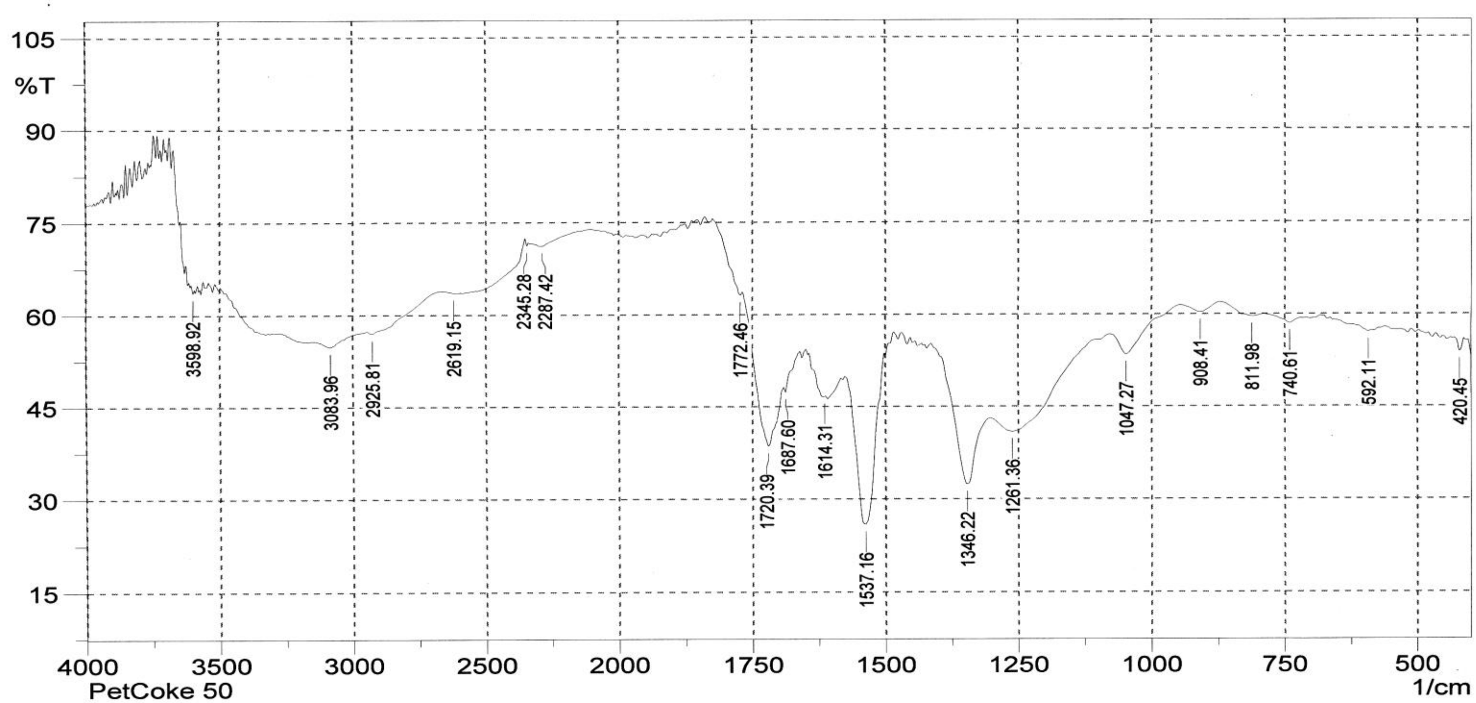
Figure 4.6: FTIR Spectrum for Calcined Coke Nitrated at 50˚C



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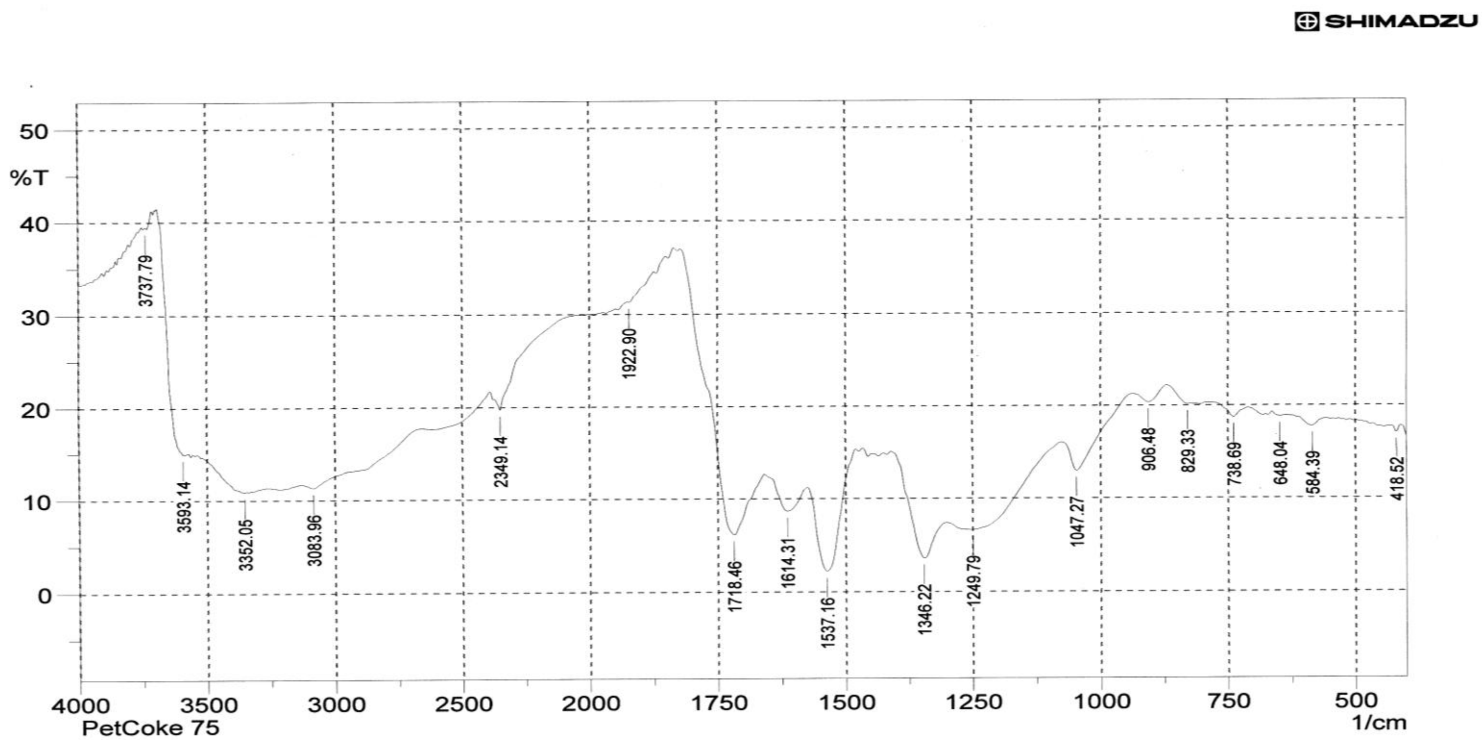
4.7: FTIR Spectrum for Calcined Coke Nitrated at 75˚C



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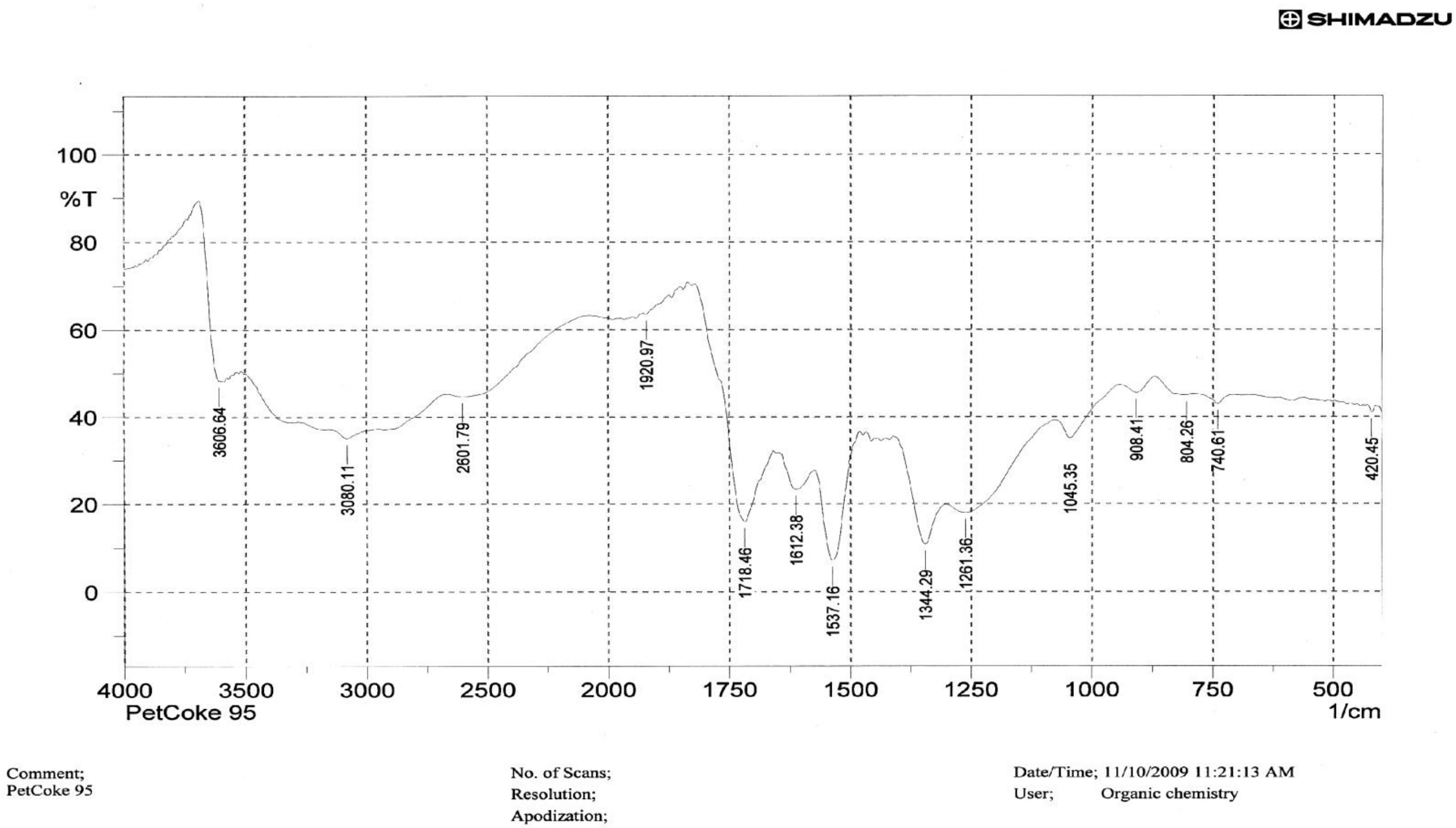
4.8: FTIR Spectrum for Calcined Coke Nitrated at 95˚C



Figure

4.1 DISCUSSIONS

4.1.1 Thermo Gravimetric Analysis (TGA)

Based on the data gained from the Thermo Gravimetric analysis, the point of evolutions of each sample are determined. The percentages of weight loss after each curve are then calculated.

Figure 4.9: Weight % vs Temperature Graph for Un-nitrated Calcined Coke

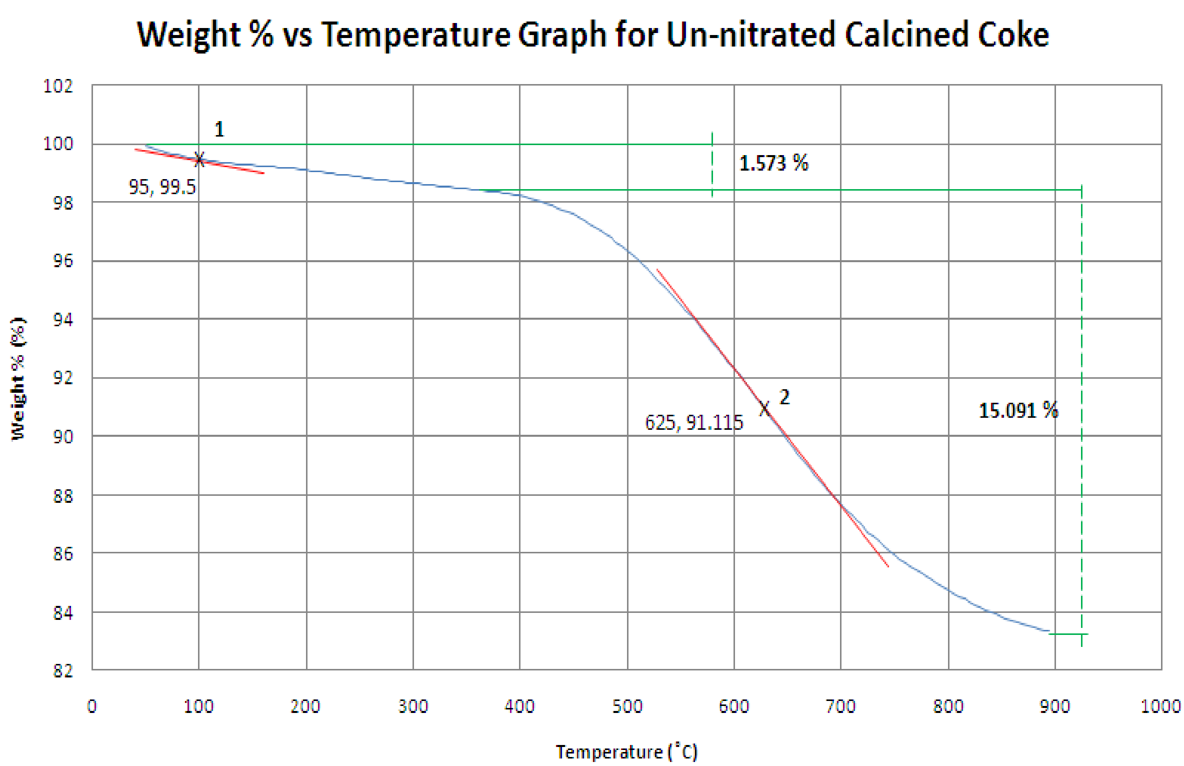


Table 4.2: Evolution Point & Weight Loss for Un-nitrated Calcined Coke Sample

Evolution	T(℃)	Weight (%)	Weight % 1	Weight % 2	Loss (%)
1	95	99.5	100	98.427	1.573
2	625	91.115	98.427	83.336	15.091

Figure 4.10: Weight % vs Temperature Graph for Calcined Coke Nitrated at 50˚C

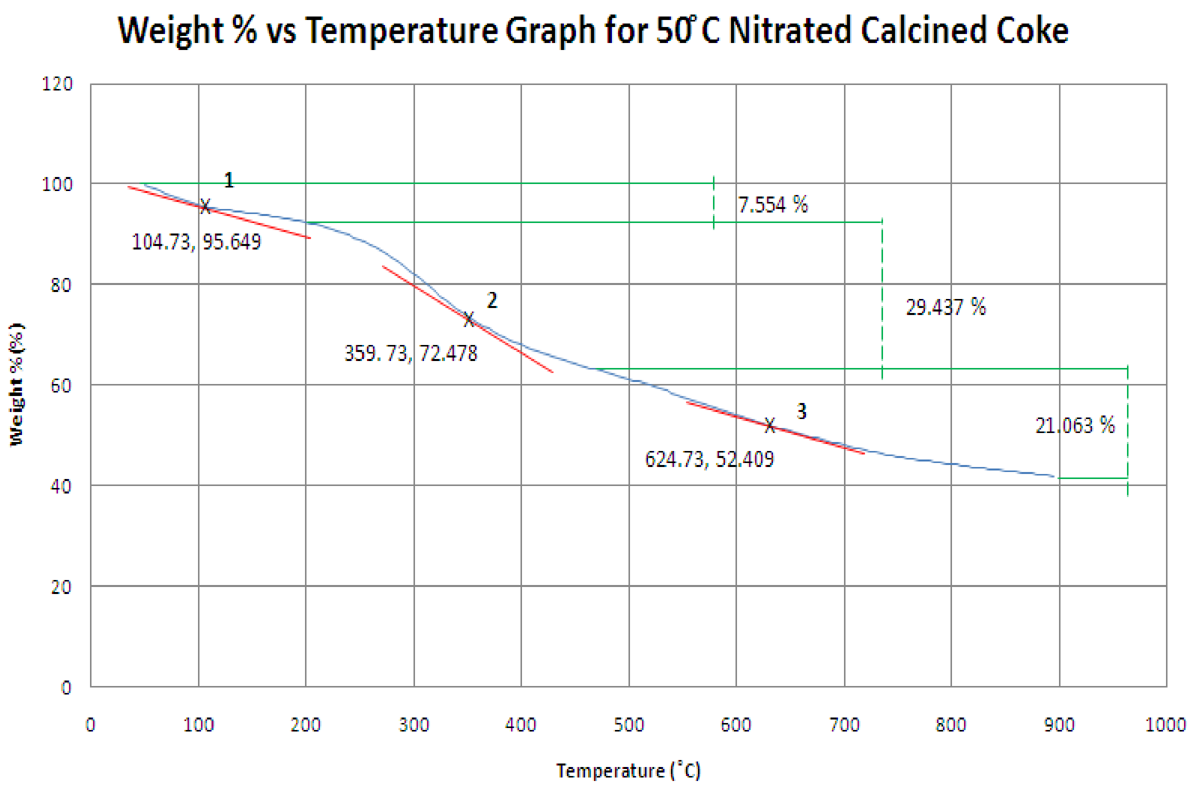


Table 4.3: Evolution Point & Weight Loss for Coke Nitrated at 50˚C

Evolution	T(℃)	Weight (%)	Weight % 1	Weight % 2	Loss (%)
1	104.73	95.649	100	92.446	7.554
2	359.73	72.478	92.446	63.009	29.437
3	624.73	52.409	63.009	41.946	21.063

Figure 4.11: Weight % vs Temperature Graph for Calcined Coke Nitrated at 75˚C

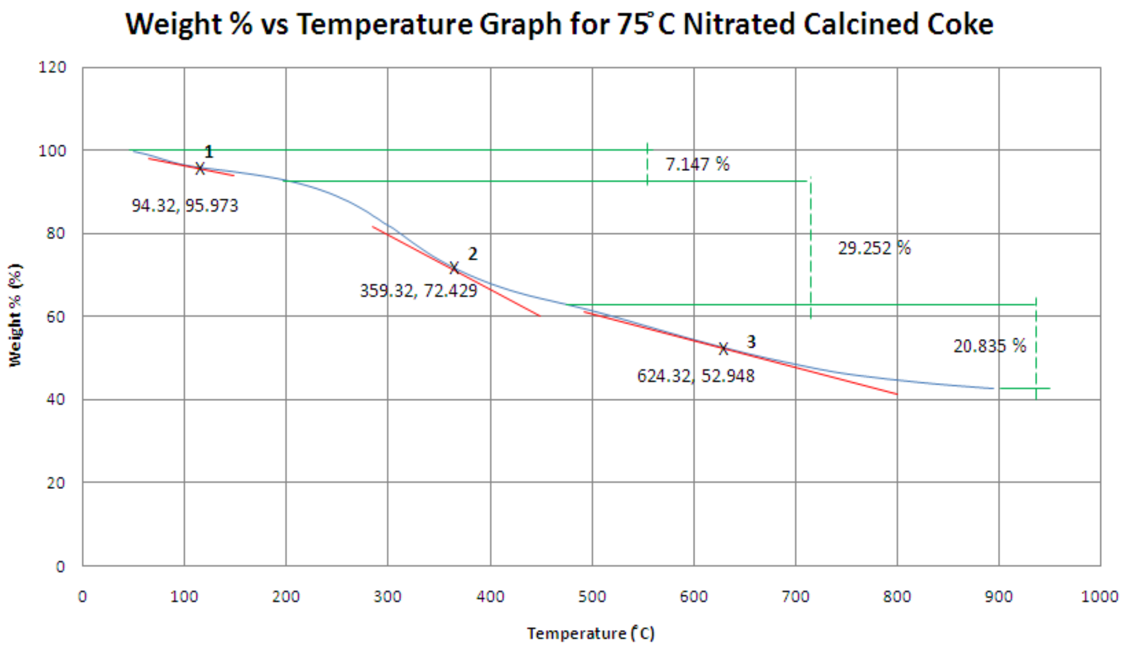


Table 4.4: Evolution Point & Weight Loss for Coke Nitrated at 75˚C

Evolution	**T(℃)**	**Weight (%)**	**Weight % 1**	**Weight % 2**	**Loss (%)**
1	94.32	95.973	100	92.853	7.147
2	359.32	72.429	92.853	63.601	29.252
3	624.32	52.948	63.601	42.766	20.835

Figure 4.12: Weight % vs Temperature Graph for Calcined Coke Nitrated at 95˚C

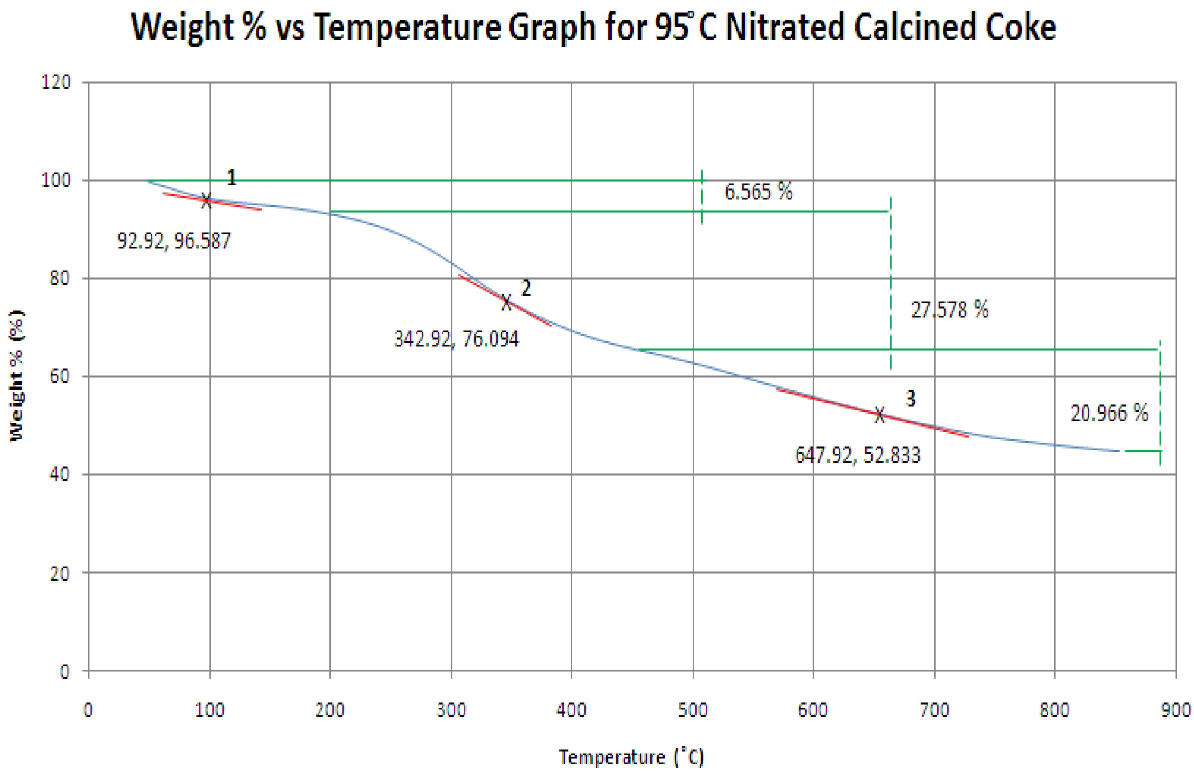


Table 4.5: Evolution Point & Weight Loss for Coke Nitrated at 95˚C

Evolution	T(˚C)	Weight (%)	Weight % 1	Weight % 2	Loss (%)
1	92.92	96.587	100	93.435	6.565
2	342.92	76.094	93.435	65.857	27.578
3	622.92	54.407	65.857	44.891	20.966

Table 4.6: Comparison of Evolution Points of Calcined Coke for Different Nitrated Temperature

	Evolution 1		Evolution 2		Evolution 3	
Sample	**Temp (˚C)**	**Weight %**	**Temp (˚C)**	**Weight %**	**Temp (˚C)**	**Weight %**
50 °C	104.73	95.649	359.73	72.478	624.73	52.409
75 °C	94.32	95.973	359.32	72.429	624.32	52.948
95 °C	92.92	96.587	342.92	76.094	622.92	54.407

Figure 4.13: Graph of Comparison for Temperature of Evolution Points of Nitrated Calcined Coke

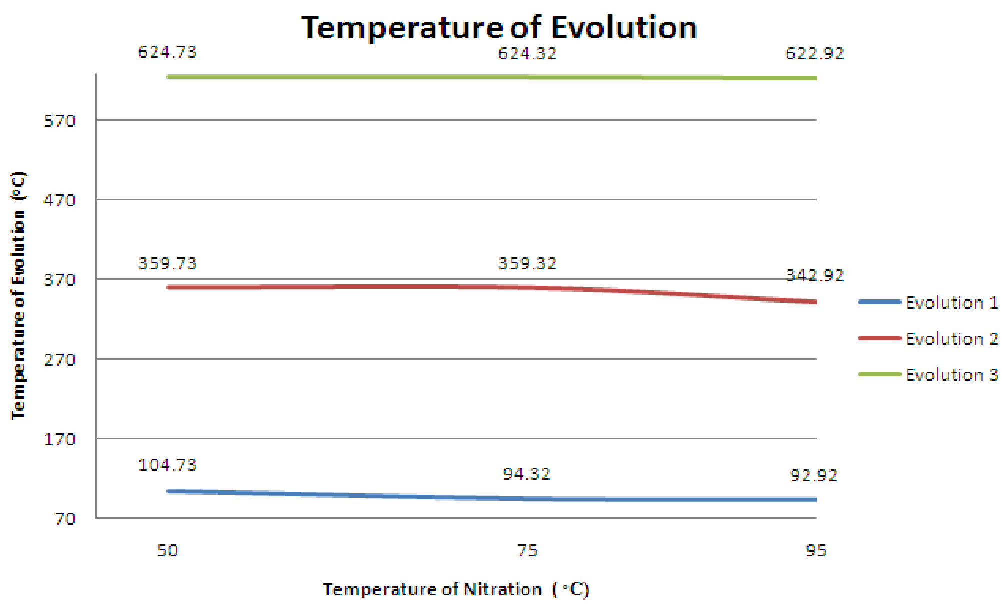


Figure 4.14: Graph of Comparison for Weight % of Evolution Points of Nitrated Calcined Coke

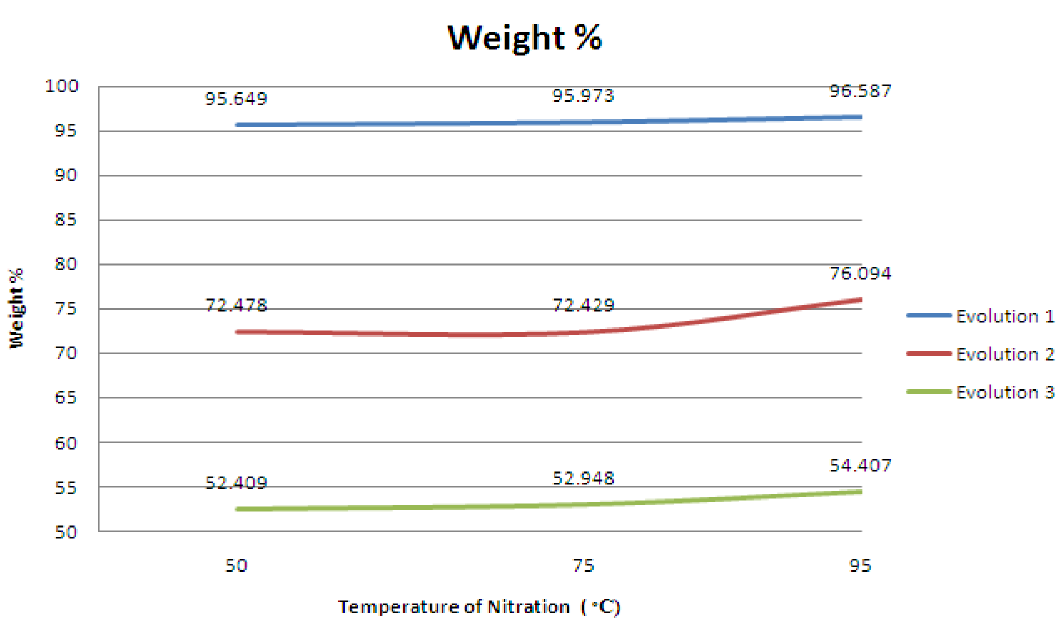
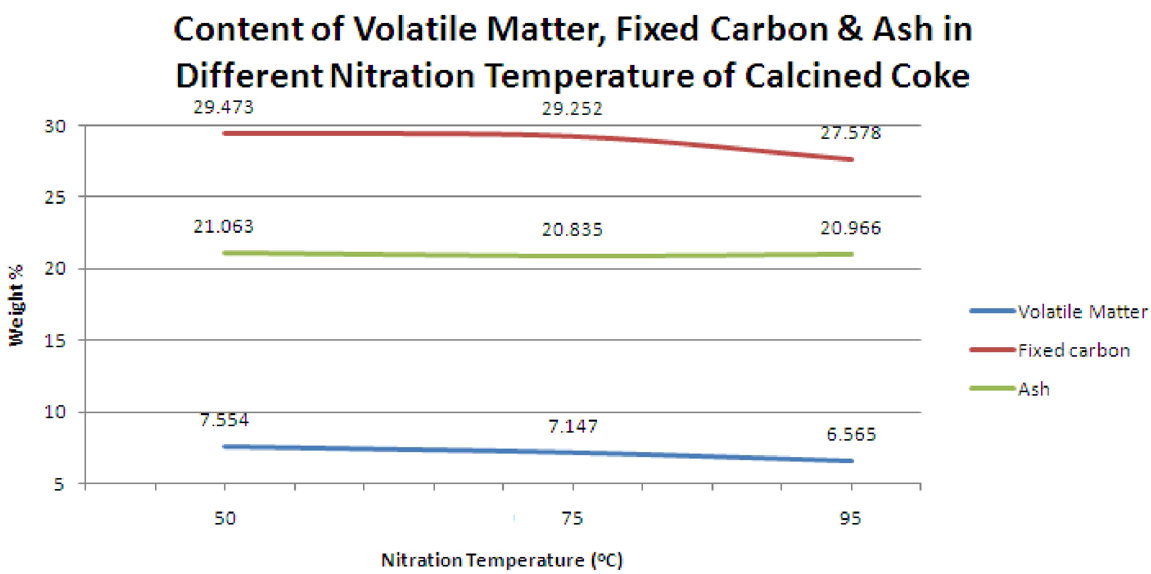


Table 4.7: Weight Percentage of Volatile Matter, Fixed carbon & Ash of the Calcined Coke Sample

Sample	Moisture / Volatile Mater (wt %)	Fixed Carbon (wt %)	Ash (wt %)
Reference	1.573	15.091	-
50˚C Nitrated	7.554	29.437	21.063
75˚C Nitrated	7.147	29.252	20.835
95˚C Nitrated	6.565	27.578	20.966

Figure 4.15: Graph of Weight Percentage of Volatile Matter, Fixed carbon & Ash of the Nitrated Calcined Coke Sample



Hard to burn fuels lose their burnable fractions (weight loss) at higher temperatures and the rate of weight loss is smaller than for easy to burn fuels. Low volatile fuels are less reactive than other fuels and therefore require special firing conditions to achieve ignition and complete burnout of the coal or coke particles [13].

From the result of thermo gravimetric analysis, it showed that compared to un-nitrated calcined coke, the nitrated calcined cokes have higher volatile matter/ moisture. However, among the three samples of nitrated calcined coke, the one nitrated at 50˚C contains the highest value of volatile matter. However, the differences are not that significant.

In term of fixed carbon content, the nitrated calcined cokes batch contain higher value of fixed carbon compare to the un-nitrated calcined coke. However, referring to Figure 4.9, we can see that the un-nitrated calcined coke lose the burnable fractions (weight loss) at higher temperature compare to nitrated calcined coke. This indicates that the un-nitrated calcined coke is rather hard to burn fuels.

Although the nitrated calcined cokes have higher fixed carbon content, they lose the burnable fractions (weight loss) or we can say the evolution points, at lower temperature compare to un-nitrated calcined coke.

The ash content for un-nitrated calcined coke cannot be determined because the evolution temperature is too high. However for nitrated calcined coke, the values are pretty much similar but the sample nitrated at 50˚C contains higher value of ash.

From the characteristics or properties gained from the analysis, we can finalized that nitrated calcined coke is more favorable than un-nitrated calcined coke for solid fuel.

4.1.2 FTIR Analysis

FTIR Analysis can identify unknown materials, determine quality or consistency of a sample and determine the amount of components in a mixture. [14]

IR absorption information is generally presented in the form of a spectrum with wavelength or wave number as the x-axis and absorption intensity or percent transmittance as the y-axis.

If the nitro group is attached to an aromatic ring, the N–O stretching bands shift to slightly down at range of wave numbers: 1550-1475 cm^{-1} and 1360-1290 cm^{-1}.

From the results, we can see that there are nitro groups attached to aromatic rings in the samples of nitrated calcined coke. At wave numbers between 1550-1475 cm^{-1} and 1360-1290 cm^{-1} there are obvious stretching bands while there is no stretching bands at similar wave numbers in un-nitrated calcined coke sample.

Table 4.8: Data for FTIR Analysis of Reference Sample (Un-treated Calcined Coke)

Peak	**Wave number, 1/λ (cm^{-1})**	**% Transmittance**
1	3465.84	84.027
2	3031.89	53.960
3	2952.81	52.168
4	2912.31	50.973
5	2848.67	51.053
6	2376.14	52.965
7	2345.28	53.164
8	1579.59	53.960
9	1438.8	53.164
10	1373.22	53.363
11	1317.29	53.562
12	1029.92	58.739
13	865.98	58.938
14	808.12	59.934
15	744.47	61.327
16	420.45	67.102

Table 4.9: Data for FTIR Analysis of Calcined Coke Nitrated at 50˚C

Peak	Wave number, 1/λ (cm^{-1})	% Transmittance
1	3598.92	64.557
2	3083.96	54.684
3	2925.81	56.962
4	2619.15	63.797
5	2345.28	71.392
6	2287.42	71.013
7	1772.46	63.418
8	1720.39	38.734
9	1687.6	47.468
10	1614.31	46.709
11	1537.16	26.582
12	1346.22	32.658
13	1261.36	41.013
14	1047.27	53.544
15	908.41	60.380
16	811.98	59.241
17	740.61	58.481
18	592.11	56.962
19	420.45	53.924

Table 4.10: Data for FTIR Analysis of Calcined Coke Nitrated at 75˚C

Peak	Wave number, 1/λ (cm^{-1})	% Transmittance
1	3737.39	41.750
2	3593.14	16.000
3	3352.05	11.750
4	3083.96	12.000
5	2349.14	21.000
6	1922.9	33.000
7	1718.46	6.750
8	1614.31	9.000
9	1537.16	2.500
10	1346.22	4.000
11	1249.79	7.000
12	1047.27	13.750
13	906.48	21.500
14	829.33	21.250
15	738.69	19.750
16	648.04	19.750
17	584.39	18.500
18	418.52	18.000

Table 4.11: Data for FTIR Analysis of Calcined Coke Nitrated at 95˚C

Peak	Wave number, 1/λ (cm^{-1})	% Transmittance
1	3606.64	47.901
2	3080.11	35.062
3	2601.79	44.444
4	1920.97	63.210
5	1718.46	15.802
6	1612.38	23.704
7	1537.16	6.914
8	1344.29	10.864
9	1261.36	18.272
10	1045.35	34.568
11	908.41	44.938
12	804.26	44.444
13	740.61	42.963
14	420.45	40.988

Figure 4.16: % Transmittance at Nitro-Group Wave Numbers for Nitrated Calcined Coke

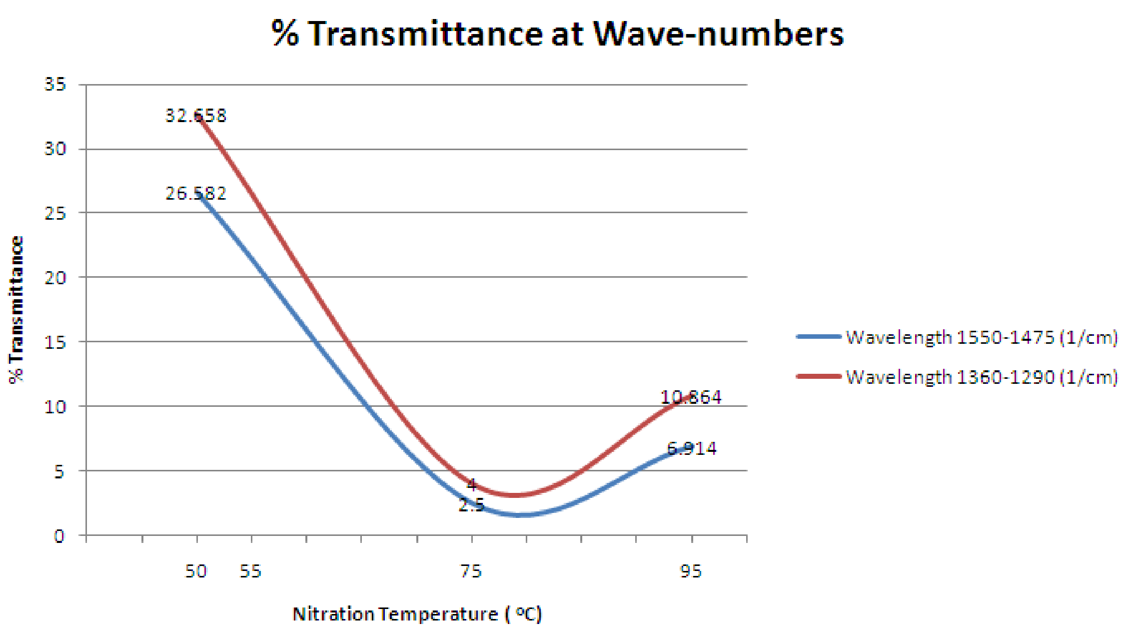
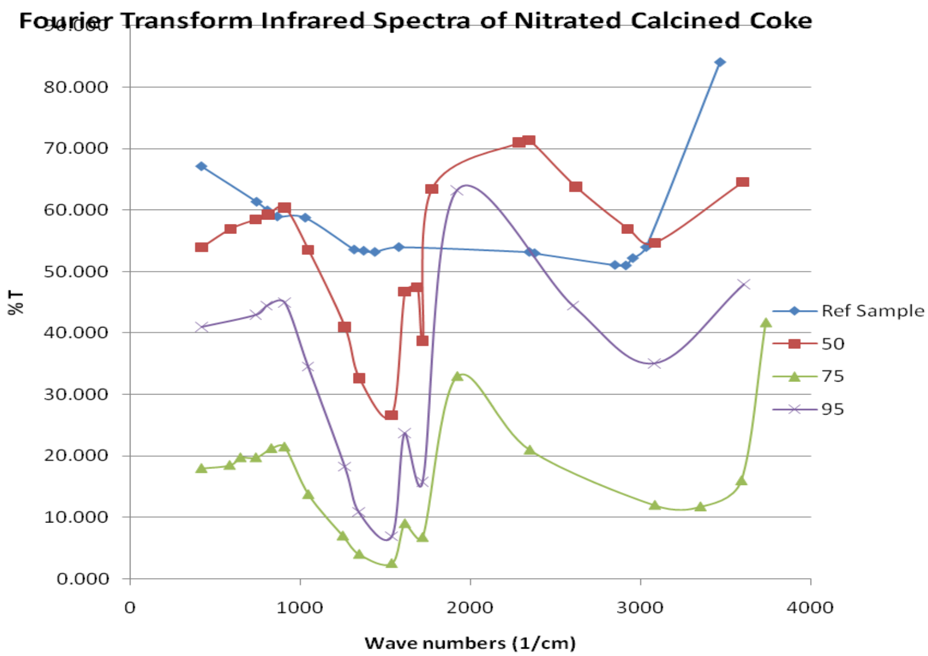


Figure 4.17: Fourier Transform Infrared Spectra of Nitrated Calcined Coke



Based on Figure 4.17, we can see that the percentage of transmittance of nitro-group wave numbers are not directly proportional to the temperature on nitration because nitration temperature of 75 ℃ gives lower percentage of transmittance of nitro-group wave numbers while nitration temperature of 50 ℃ gives the higher percentage of transmittance of nitro-group wave numbers.

Table 4.12: Comparison of Contents of Un-Nitrated & Nitrated Calcined Petroleum Coke

Properties	Calcined Petroleum Coke	Nitrated Calcined Coke		
		50˚C	75˚C	95˚C
Volatile Matter (wt %)	1.573	7.554	7.147	6.565
Fixed Carbon (wt %)	15.091	29.437	29.252	27.578
Ash Content (wt %)	-	21.063	20.835	20.966
Nitro Group (%T)	-	26.582 / 32.658	2.5 / 4.0	6.914 / 10.864

Table 4.13 : Comparison Between Water Dispersible Solid Fuel and Nitrated Petroleum Coke Properties

Properties	Water Dispersible Solid Fuel Compatible	Nitrated Calcined Coke		
		50˚C	**75˚C**	**95˚C**
Volatile Matter (wt %)	>18	7.554	7.147	6.565
Fixed Carbon (wt %)	< 9	29.437	29.252	27.578
Ash Content (wt %)	<0.25	21.063	20.835	20.966
Nitro Group (%T)	Available	26.582 / 32.658	2.5 /4.0	6.914 / 10.864

For the nitrated to be compatible as water dispersible solid fuel, the volatile matter must be higher than 18 % of its weight, while the fixed carbon content should be lower than 9% of its weight. Other than that, the ash content must be lower than 0.25% of its weight and obviously, to be water dispersible, there must be nitro-group attached to the aromatic ring in the nitrated calcined coke.

Comparing the mentioned properties, we can see that the nitrated calcined coke didn't meet the expectations in term of volatile matter, fixed carbon & ash content. However, since there is nitro-group attached, the calcined coke is now more soluble than before the nitration treatment.

4.2 CONCLUSIONS

As a conclusion, the methodology which is used in this project succeed in achieving the objectives of the whole project which are to determine if the calcined coke can be water dispersible, to study the improvements of calcined coke after treating with nitration, and to compare the characteristics of solid fuel and nitrated calcined coke to see if there is a possibility for the coke to be developed to water dispersible solid fuel. From the analysis we found that the calcined coke can be dispersible. The properties of the calcined coke did improved after the nitration however not to the extent of compatible to be solid fuel.

RECOMMENDATION

Widened the study of the experiment involved in this project by adding more variety to the experiment such as adding more nitration temperature to compare the results and determine which temperature is the optimum temperature for nitration of calcined coke.

Use different nitration period to study the effect of time to the nitration process of the calcined coke.

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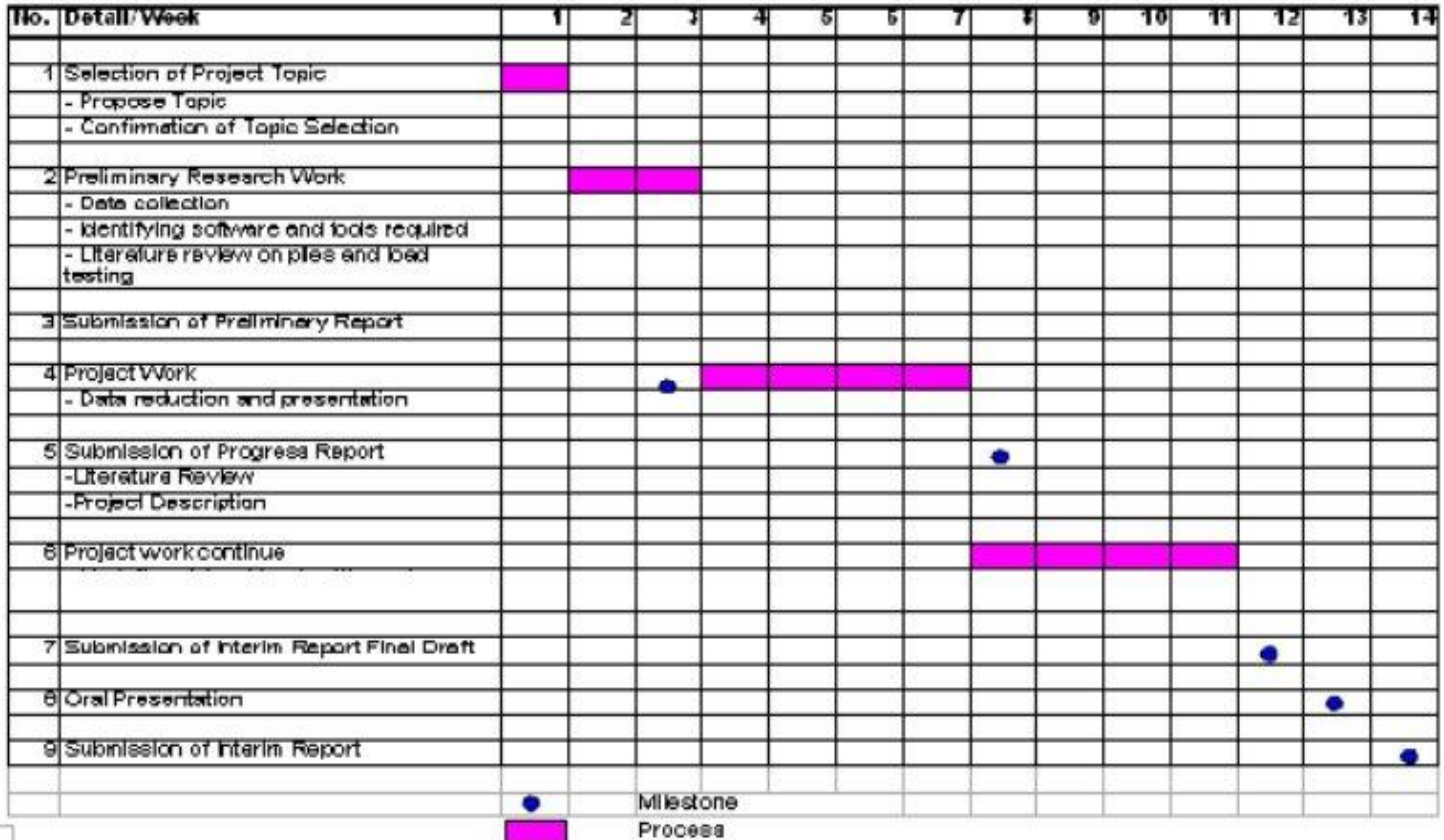
APPENDIXES

I. Gantt Chart FYP 1



II. Gantt Chart FYP 2

No.	Detail/ Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Project Work Continue														
	-Practical/Laboratory Work														
2	Submission of Progress Report 1			●											
3	Project Work Continue														
	-Practical/Laboratory Work														
4	Submission of Progress Report 2								●						
5	Project work continue														
	-Practical/Laboratory Work														
6	Submission of Dissertation Final Draft												●		
7	Oral Presentation													●	
8	Submission of Project Dissertation														●

● Suggested milestone

Process

III. Thermo Gravimetric Analysis Data

Reference Sample (Untreated Calcined Coke)

Temperature (°C)	Weight % (%)	Temperature (°C)	Weight % (%)	Temperature (°C)	Weight % (%)
49.99	99.94	159.99	99.242	269.99	98.772
54.99	99.881	164.99	99.227	274.99	98.743
59.99	99.823	169.99	99.212	279.99	98.726
64.99	99.765	174.99	99.199	284.99	98.704
69.99	99.712	179.99	99.179	289.99	98.684
74.99	99.661	184.99	99.167	294.99	98.664
79.99	99.617	189.99	99.149	299.99	98.643
84.99	99.575	194.99	99.129	304.99	98.625
89.99	99.534	199.99	99.106	309.99	98.606
94.99	99.5	204.99	99.085	314.99	98.587
99.99	99.467	209.99	99.062	319.99	98.568
104.99	99.436	214.99	99.039	324.99	98.551
109.99	99.406	219.99	99.015	329.99	98.532
114.99	99.384	224.99	98.992	334.99	98.514
119.99	99.363	229.99	98.969	339.99	98.498
124.99	99.349	234.99	98.941	344.99	98.481
129.99	99.33	239.99	98.919	349.99	98.463
134.99	99.313	244.99	98.894	354.99	98.445
139.99	99.299	249.99	98.868	359.99	98.427
144.99	99.288	254.99	98.844	364.99	98.406
149.99	99.272	259.99	98.817	369.99	98.386
154.99	99.257	264.99	98.796	374.99	98.362

Temperature (°C)	Weight % (%)	Temperature (°C)	Weight % (%)	Temperature (°C)	Weight % (%)
379.99	98.341	504.99	96.203	629.32	52.633
384.99	98.315	509.99	96.029	634.32	52.319
389.99	98.293	514.99	95.88	639.32	52.003
394.99	98.264	519.99	95.674	644.32	51.695
399.99	98.228	524.99	95.479	649.32	51.386
404.99	98.18	529.99	95.315	654.32	51.091
409.99	98.127	534.99	95.105	659.32	50.795
414.99	98.067	539.99	94.882	664.32	50.51
419.99	98.011	544.99	94.689	669.32	50.222
424.99	97.94	549.99	94.502	674.32	49.937
429.99	97.872	554.99	94.314	679.32	49.669
434.99	97.805	559.99	94.126	684.32	49.407
439.99	97.738	564.99	93.913	689.32	49.142
444.99	97.67	569.99	93.674	694.32	48.894
449.99	97.597	574.99	93.448	699.32	48.636
454.99	97.475	579.99	93.217	704.32	48.368
459.99	97.371	584.99	92.972	709.32	48.105
464.99	97.272	589.99	92.753	714.32	47.857
469.99	97.17	594.99	92.532	719.32	47.61
474.99	97.021	599.99	92.316	724.32	47.372
479.99	96.922	604.99	92.088	729.32	47.145
484.99	96.807	609.99	91.859	734.32	46.924
489.99	96.65	614.99	91.607	739.32	46.713
494.99	96.519	619.99	91.359	744.32	46.502
499.99	96.355	624.99	91.115	749.32	46.319

Temperature (°C)	Weight % (%)	Temperature (°C)	Weight % (%)
754.99	85.811	829.99	84.164
759.99	85.68	834.99	84.081
764.99	85.554	839.99	84.000
769.99	85.429	844.99	83.922
774.99	85.308	849.99	83.849
779.99	85.185	854.99	83.78
784.99	85.07	859.99	83.715
789.99	84.96	864.99	83.652
794.99	84.85	869.99	83.593
799.99	84.747	874.99	83.536
804.99	84.643	879.99	83.482
809.99	84.541	884.99	83.431
814.99	84.441	889.99	83.383
819.99	84.346	894.99	83.336
824.99	84.253		

Calcined Coke Nitrated at 50˚C

Temperature (°C)	Weight % (%)	Temperature (°C)	Weight % (%)	Temperature (°C)	Weight % (%)
49.73	99.72	164.73	93.777	279.73	85.388
54.73	99.387	169.73	93.614	284.73	84.663
59.73	99.036	174.73	93.45	289.73	83.894
64.73	98.644	179.73	93.27	294.73	83.096
69.73	98.227	184.73	93.085	299.73	82.263
74.73	97.799	189.73	92.886	304.73	81.399
79.73	97.377	194.73	92.668	309.73	80.482
84.73	96.966	199.73	92.446	314.73	79.613
89.73	96.571	204.73	92.198	319.73	78.726
94.73	96.218	209.73	91.937	324.73	77.843
99.73	95.909	214.73	91.648	329.73	76.979
104.73	95.649	219.73	91.341	334.73	76.164
109.73	95.412	224.73	91.019	339.73	75.367
114.73	95.208	229.73	90.666	344.73	74.609
119.73	95.038	234.73	90.291	349.73	73.876
124.73	94.877	239.73	89.884	354.73	73.177
129.73	94.738	244.73	89.434	359.73	72.498
134.73	94.602	249.73	88.964	364.73	71.862
139.73	94.475	254.73	88.459	369.73	71.256
144.73	94.347	259.73	87.916	374.73	70.653
149.73	94.216	264.73	87.344	379.73	70.091
154.73	94.076	269.73	86.727	384.73	69.555
159.73	93.929	274.73	86.078	389.73	69.032

Temperature (°C)	Weight % (%)	Temperature (°C)	Weight % (%)	Temperature (°C)	Weight % (%)
394.73	68.522	514.73	60.287	634.73	51.762
399.73	68.041	519.73	59.939	639.73	51.449
404.73	67.582	524.73	59.594	644.73	51.127
409.73	67.141	529.73	59.234	649.73	50.819
414.73	66.716	534.73	58.875	654.73	50.523
419.73	66.318	539.73	58.505	659.73	50.223
424.73	65.942	544.73	58.146	664.73	49.937
429.73	65.571	549.73	57.782	669.73	49.654
434.73	65.212	554.73	57.412	674.73	49.369
439.73	64.883	559.73	57.05	679.73	49.098
444.73	64.559	564.73	56.689	684.73	48.839
449.73	64.252	569.73	56.326	689.73	48.569
454.73	63.953	574.73	55.962	694.73	48.319
459.73	63.665	579.73	55.602	699.73	48.06
464.73	63.381	584.73	55.239	704.73	47.8
469.73	63.099	589.73	54.873	709.73	47.538
474.73	62.802	594.73	54.504	714.73	47.284
479.73	62.512	599.73	54.136	719.73	47.035
484.73	62.217	604.73	53.774	724.73	46.803
489.73	61.919	609.73	53.417	729.73	46.573
494.73	61.605	614.73	53.081	734.73	46.352
499.73	61.283	619.73	52.74	739.73	46.142
504.73	60.961	624.73	52.409	744.73	45.931
509.73	60.629	629.73	52.082	749.73	45.753

Temperature (°C)	Weight % (%)	Temperature (°C)	Weight % (%)
754.73	45.582	829.73	43.43
759.73	45.416	834.73	43.304
764.73	45.253	839.73	43.183
769.73	45.098	844.73	43.065
774.73	44.947	849.73	42.945
779.73	44.799	854.73	42.831
784.73	44.657	859.73	42.716
789.73	44.509	864.73	42.608
794.73	44.376	869.73	42.498
799.73	44.24	874.73	42.388
804.73	44.101	879.73	42.277
809.73	43.965	884.73	42.166
814.73	43.83	889.73	42.055
819.73	43.697	894.73	41.946
824.73	43.559		

Calcined Coke Nitrated at 75˚C

Temperature (°C)	Weight % (%)	Temperature (°C)	Weight % (%)	Temperature (°C)	Weight % (%)
49.32	99.811	164.32	94.367	279.32	85.212
54.32	99.552	169.32	94.182	284.32	84.362
59.32	99.277	174.32	93.995	289.32	83.635
64.32	98.96	179.32	93.8	294.32	82.741
69.32	98.601	184.32	93.574	299.32	82.012
74.32	98.216	189.32	93.355	304.32	81.34
79.32	97.836	194.32	93.108	309.32	80.431
84.32	97.466	199.32	92.853	314.32	79.536
89.32	97.124	204.32	92.577	319.32	78.615
94.32	96.834	209.32	92.276	324.32	77.74
99.32	96.581	214.32	91.966	329.32	76.883
104.32	96.353	219.32	91.627	334.32	76.053
109.32	96.148	224.32	91.263	339.32	75.255
114.32	95.973	229.32	90.879	344.32	74.477
119.32	95.806	234.32	90.453	349.32	73.772
124.32	95.652	239.32	90.018	354.32	73.084
129.32	95.499	244.32	89.538	359.32	72.429
134.32	95.357	249.32	89.012	364.32	71.787
139.32	95.206	254.32	88.467	369.32	71.182
144.32	95.05	259.32	87.913	374.32	70.612
149.32	94.892	264.32	87.295	379.32	70.06
154.32	94.727	269.32	86.634	384.32	69.545
159.32	94.551	274.32	85.944	389.32	69.028

Temperature (°C)	**Weight % (%)**	**Temperature (°C)**	**Weight % (%)**	**Temperature (°C)**	**Weight % (%)**
394.32	68.542	514.32	60.546	634.32	52.319
399.32	68.08	519.32	60.2	639.32	52.003
404.32	67.638	524.32	59.85	644.32	51.695
409.32	67.218	529.32	59.491	649.32	51.386
414.32	66.806	534.32	59.141	654.32	51.091
419.32	66.415	539.32	58.787	659.32	50.795
424.32	66.046	544.32	58.434	664.32	50.51
429.32	65.697	549.32	58.072	669.32	50.222
434.32	65.372	554.32	57.71	674.32	49.937
439.32	65.052	559.32	57.35	679.32	49.669
444.32	64.748	564.32	57	684.32	49.407
449.32	64.455	569.32	56.648	689.32	49.142
454.32	64.172	574.32	56.287	694.32	48.894
459.32	63.882	579.32	55.939	699.32	48.636
464.32	63.601	584.32	55.599	704.32	48.368
469.32	63.324	589.32	55.254	709.32	48.105
474.32	63.037	594.32	54.907	714.32	47.857
479.32	62.746	599.32	54.575	719.32	47.61
484.32	62.445	604.32	54.245	724.32	47.372
489.32	62.143	609.32	53.918	729.32	47.145
494.32	61.834	614.32	53.586	734.32	46.924
499.32	61.516	619.32	53.266	739.32	46.713
504.32	61.196	624.32	52.948	744.32	46.502
509.32	60.869	629.32	52.633	749.32	46.319

Temperature (°C)	Weight % (%)	Temperature (°C)	Weight % (%)
754.32	46.148	829.32	44.07
759.32	45.987	834.32	43.954
764.32	45.83	839.32	43.845
769.32	45.677	844.32	43.739
774.32	45.522	849.32	43.632
779.32	45.374	854.32	43.532
784.32	45.232	859.32	43.434
789.32	45.092	864.32	43.335
794.32	44.958	869.32	43.239
799.32	44.828	874.32	43.142
804.32	44.693	879.32	43.047
809.32	44.559	884.32	42.954
814.32	44.432	889.32	42.86
819.32	44.311	894.32	42.766
824.32	44.188		

Calcined Coke Nitrated at 95˚C

Temperature (°C)	Weight % (%)	Temperature (°C)	Weight % (%)	Temperature (°C)	Weight % (%)
47.92	99.933	162.92	94.511	277.92	86.494
52.92	99.447	167.92	94.357	282.92	85.784
57.92	99.099	172.92	94.196	287.92	85.063
62.92	98.714	177.92	94.021	292.92	84.298
67.92	98.323	182.92	93.84	297.92	83.509
72.92	97.922	187.92	93.646	302.92	82.663
77.92	97.536	192.92	93.435	307.92	81.823
82.92	97.181	197.92	93.213	312.92	80.967
87.92	96.868	202.92	92.983	317.92	80.127
92.92	96.587	207.92	92.73	322.92	79.28
97.92	96.344	212.92	92.451	327.92	78.437
102.92	96.136	217.92	92.163	332.92	77.637
107.92	95.949	222.92	91.852	337.92	76.858
112.92	95.787	227.92	91.513	342.92	76.094
117.92	95.65	232.92	91.155	347.92	75.365
122.92	95.517	237.92	90.765	352.92	74.668
127.92	95.392	242.92	90.345	357.92	73.996
132.92	95.276	247.92	89.889	362.92	73.353
137.92	95.162	252.92	89.417	367.92	72.736
142.92	95.041	257.92	88.913	372.92	72.15
147.92	94.922	262.92	88.353	377.92	71.583
152.92	94.792	267.92	87.76	382.92	71.051
157.92	94.661	272.92	87.138	387.92	70.518

Temperature (°C)	Weight % (%)	Temperature (°C)	Weight % (%)	Temperature (°C)	Weight % (%)
392.92	70.018	512.92	62.016	632.92	53.781
397.92	69.556	517.92	61.677	637.92	53.459
402.92	69.1	522.92	61.332	642.92	53.145
407.92	68.657	527.92	60.968	647.92	52.833
412.92	68.237	532.92	60.61	652.92	52.544
417.92	67.843	537.92	60.254	657.92	52.25
422.92	67.466	542.92	59.903	662.92	51.957
427.92	67.105	547.92	59.544	667.92	51.667
432.92	66.775	552.92	59.184	672.92	51.386
437.92	66.451	557.92	58.826	677.92	51.101
442.92	66.147	562.92	58.475	682.92	50.831
447.92	65.857	567.92	58.12	687.92	50.567
452.92	65.572	572.92	57.771	692.92	50.309
457.92	65.293	577.92	57.419	697.92	50.057
462.92	65.019	582.92	57.064	702.92	49.791
467.92	64.737	587.92	56.722	707.92	49.537
472.92	64.459	592.92	56.384	712.92	49.274
477.92	64.177	597.92	56.046	717.92	49.024
482.92	63.883	602.92	55.706	722.92	48.781
487.92	63.589	607.92	55.375	727.92	48.547
492.92	63.28	612.92	55.058	732.92	48.327
497.92	62.974	617.92	54.726	737.92	48.106
502.92	62.667	622.92	54.407	742.92	47.9
507.92	62.341	627.92	54.091	747.92	47.703

Temperature (°C)	**Weight % (%)**
752.92	47.532
757.92	47.364
762.92	47.206
767.92	47.05
772.92	46.896
777.92	46.747
782.92	46.603
787.92	46.46
792.92	46.324
797.92	46.191
802.92	46.063
807.92	45.928
812.92	45.803
817.92	45.675
822.92	45.551
827.92	45.431
832.92	45.316
837.92	45.205
842.92	45.101
847.92	44.999
852.92	44.891

IV. FTIR Analysis Data

<u>Data for FTIR Analysis of Reference Sample (Un-treated Calcined Coke</u>

No.	Peak	Height	Corr. Height	Base (H)	Base (L)	Area	Corr. Area
1	420.45	32.857	2.739	437.81	412.74	4.053	0.16
2	744.47	38.635	2.329	769.54	676.97	18.099	0.577
3	808.12	40.074	1.209	825.48	771.47	11.639	0.248
4	865.98	41.139	1.907	908.41	837.05	15.868	0.471
5	1029.92	41.236	0.757	1051.13	991.34	13.599	0.14
6	1317.29	46.383	0.667	1332.72	1220.86	29.493	0.188
7	1373.22	46.58	0.755	1384.79	1369.37	4.112	0.062
8	1438.8	47.007	0.652	1444.58	1431.08	3.691	0.039
9	1579.59	46.145	1.61	1631.67	1571.88	14.596	0.726
10	2345.28	46.942	0.784	2352.99	2337.56	4.195	0.046
11	2376.14	47.114	0.743	2393.5	2352.99	11.062	0.122
12	2848.67	48.977	0.691	2877.6	2758.02	26.565	0.176
13	2912.31	49.098	1	2943.17	2877.6	18.965	0.274
14	2952.81	47.786	0.349	2987.53	2945.1	11.736	0.049
15	3031.89	46.249	0.45	3415.7	3026.1	73.085	4.728
16	3465.84	16.485	0.088	3467.77	3452.34	1.187	0.003

Data for FTIR Analysis of Calcined Coke Nitrated at 50˚C

No.	Peak	Height	Corr. Height	Base (H)	Base (L)	Area	Corr. Area
1	420.45	46.118	2.145	430.1	412.74	4.506	0.139
2	592.11	42.881	0.377	621.04	586.32	8.304	0.034
3	740.61	41.505	1.007	786.9	721.33	14.921	0.192
4	811.98	40.393	0.333	827.41	788.83	8.623	0.048
5	908.41	39.692	1.408	945.05	873.69	15.32	0.362
6	1047.27	46.518	4.297	1076.21	946.98	31.055	1.517
7	1261.36	59.132	4.811	1301.86	1103.21	65.943	4.672
8	1346.22	67.578	15.369	1413.72	1303.79	40.83	6.489
9	1537.16	74.105	25.87	1573.81	1502.44	30.886	10.458
10	1614.31	53.375	0.462	1633.59	1612.38	6.824	0.174
11	1687.6	52.661	1.546	1691.46	1660.6	9.011	0.072
12	1720.39	61.448	15.434	1768.6	1691.46	25.402	5.49
13	1772.46	36.797	1.327	1824.53	1768.6	8.963	0.089
14	2287.42	28.791	0.075	2291.28	2142.77	20.666	0.007
15	2345.28	28.739	0.778	2352.99	2339.49	1.947	0.028
16	2619.15	36.4	0.057	2667.37	2613.37	10.556	0.008
17	2952.81	43.013	0.816	2945.1	2669.3	60.938	1
18	3083.96	45.187	0.966	3120.61	3039.6	20.857	0.34
19	3598.92	36.363	0.974	3606.64	3593.14	2.601	0.045

Data for FTIR Analysis of Calcined Coke Nitrated at 75˚C

No.	Peak	Height	Corr. Height	Base (H)	Base (L)	Area	Corr. Area
1	418.52	82.963	0.728	430.1	410.81	14.623	0.148
2	584.39	82.262	0.935	622.96	559.32	46.983	0.639
3	648.04	81.192	0.269	663.47	636.47	19.534	0.115
4	738.69	81.279	1.198	790.76	711.68	55.931	0.684
5	829.33	79.861	0.204	867.91	825.48	28.796	0.133
6	906.48	79.646	1.297	935.41	869.84	44.382	0.889
7	1047.27	87.055	4.11	1074.28	937.34	106.204	5.867
8	1249.79	93.386	2.707	1299.93	1076.21	229.283	14.433
9	1346.22	96.414	6.955	1409.87	1301.86	123.393	17.747
10	1537.16	97.817	10.64	1573.81	1481.23	110.565	28.943
11	1614.31	91.356	3.217	1656.74	1575.73	80.334	5.322
12	1718.46	93.889	15.517	1820.68	1658.67	137.383	29.289
13	1922.9	68.708	0.263	1926.75	1874.68	25.24	0.144
14	2349.14	80.292	1.843	2370.35	2071.41	174.617	0.807
15	3083.96	88.692	0.697	3126.4	2937.38	172.919	1.51
16	3352.05	89.128	0.113	3375.2	3344.34	29.656	0.101
17	3593.14	85.015	0.539	3691.5	3591.21	63.688	3.225
18	3737.79	60.589	0.027	3739.72	3735.86	1.559	0.001

Data for FTIR Analysis of Calcined Coke Nitrated at 95˚C

No.	Peak	Height	Corr. Height	Base (H)	Base (L)	Area	Corr. Area
1	420.45	59.028	1.596	430.1	412.74	6.568	0.131
2	740.61	57.07	2.189	790.76	707.83	29.243	0.589
3	804.26	55.055	0.864	869.84	792.69	25.934	0.714
4	908.41	54.5	2.698	941.2	871.76	22.851	0.886
5	1045.35	64.95	6.079	1076.21	943.13	51.861	3.326
6	1261.36	82.008	5.22	1299.93	1078.13	131.344	9.499
7	1344.29	89.206	15.19	1409.87	1301.86	76.778	14.429
8	1537.16	92.904	24.127	1573.81	1481.23	69.366	23.472
9	1612.38	76.71	6.474	1647.1	1575.73	41.82	4.068
10	1718.46	84.113	30.392	1820.68	1658.67	77.44	24.913
11	1920.97	36.45	0.846	1928.68	1874.68	9.938	0.144
12	2601.79	55.354	2.685	2667.37	2096.48	161.705	7.941
13	3080.11	64.972	2.036	3139.9	2993.32	64.798	1.588
14	3606	51.665	2.744	3689.57	3600.85	17.191	1.064